High Temperature Superconductors: Crystal Growth and Characterization

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Background

The study of high transition-temperature (Tc) superconductors - materials which exhibit zero electrical resistance and the Meissner effect (perfect diamagnetism) at temperatures significantly higher than conventional superconductors - has been one of the most intensely studied areas of condensed matter physics for nearly 30 years [1]. Our group explores a class of materials called 'cuprates,' whose crystal structure is partially made up of CuO_2 planes which are thought to be the key to understanding the unknown mechanism behind the behavior of these materials. However, it is difficult to analyze the true electrical behavior of the CuO_2 planes, as many of their important effects tend to be masked by the random disorder inherent in solids.

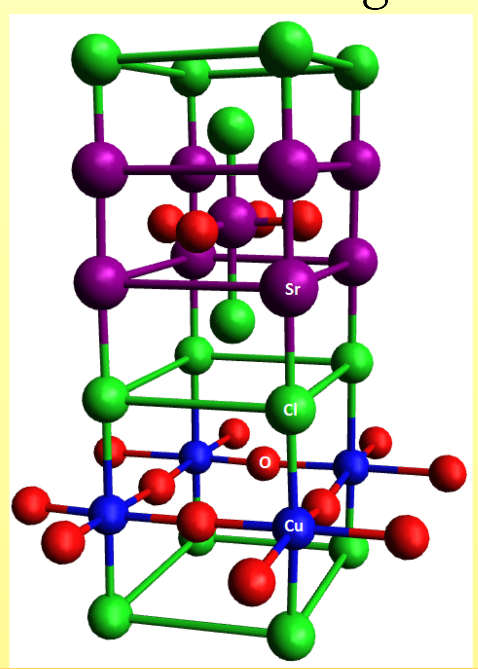

Figure 1: Crystal structure of $Sr_2CuO_2Cl_2$

Material Preparation

Our group focuses heavily on the growth of pure, homogenous single-crystal cuprates, in order to study samples that are largely free from disorder stemming from impurities. This process begins with a very precise ratio of various metal powders, for example Sm_2O_3, CeO_3 and CuO, where specific amounts of CeO_2 are used to 'dope' the material, introducing either conduction electrons or holes into the structure of the crystal. Doping allows for control of the specific electronic properties to be measured later on, with a specific doping level exhibiting 'optimal' behavior. This mixture is ground and baked several times before becoming homogenous enough to produce a pure single crystal, much like the one seen in figure 3.

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Crystal Growth

Growing a crystal requires a polycrystalline 'feed' rod, made from the previously prepared powder mixture, and an already grown 'seed' crystal from a previous growth. The feed rod is melted onto the seed crystal, as seen in figure 2, while the rods are slowly pulled apart to allow for vertical growth. The growth takes place at a rate of about 0.25 mm/hour in a travelling-solvent floating-zone (TSFZ) furnace over the course of several days. The growth temperature required depends on the melting points of the desired crystal and the CuO 'flux' which counteracts the incongruent melting of the compound.

Figure 2: Image of a growth in progress in a TSFZ furnace. The feed rod is seen on stop, with the molten zone in the middle, the seed crystal on the bottom, and the lamp filaments in the background.

Figure 3: Completed single crystal just after a finished growth. This particular example is approximately 5 cm in length from top to bottom.

Characterization

Once a growth is finished, the new crystal must be characterized in order to determine the orientation of the CuO_2 planes for later measurements. This is done using the Laue X-Ray Diffraction technique, typical results of which are shown in figure 4. A symmetrical, sharp diffraction pattern indicates that the sample is a pure, single crystal being viewed from the CuO_2 planes. From here, the samples are annealed in Argon gas at temperatures above 900° C for several days to reduce extraneous oxygen atoms introduced into the crystal structure during the growth. The annealing process is continuously refined by observing how it affects the relevant superconducting properties of the material.

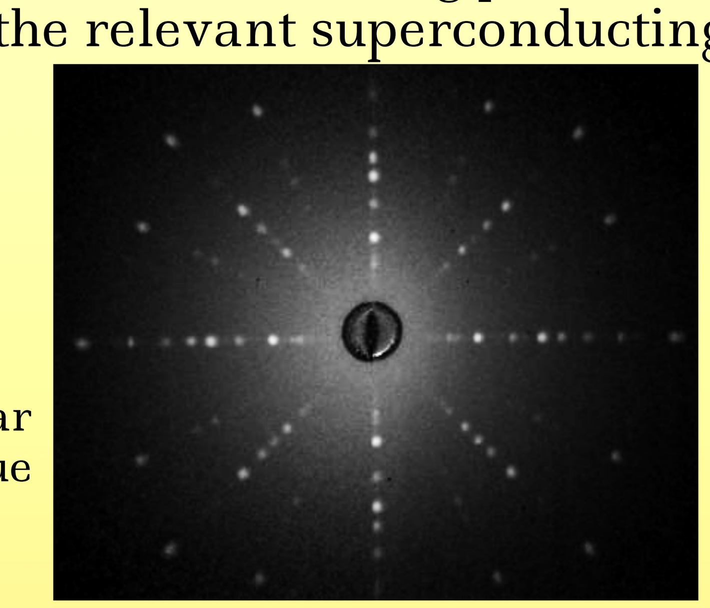

Figure 4: An example of near ideal results of Laue X-ray diffraction.

Measurements and Results

The annealing process is evaluated by subjecting the sample to various magnetic fields and temperatures in a Magnetic Properties Measurement System (MPMS.) An example of this measurement is seen in figure 5. The main property desired is the T_C of the sample, which is indicated by the sharp transition around 18 K. Thus, the annealing process is optimized for each compound to obtain the highest T_C and sharpest transition possible. The electronic resistivity properties of the materials are then determined through DC transport measurement to further measure the T_C as well as other doping dependent properties.

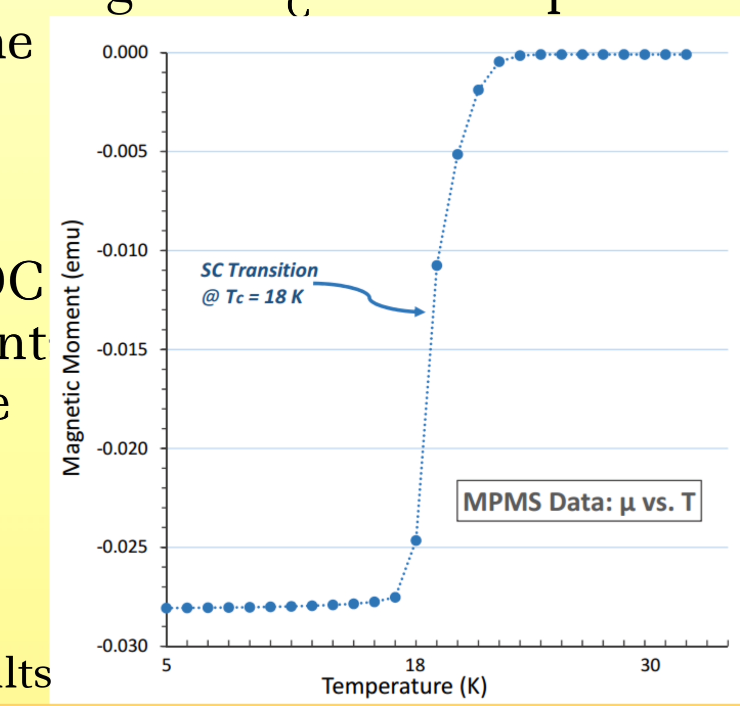

Figure 5: MPMS results

Next Steps

Once pure single crystal samples have been grown and characterized, the groundwork is in place for further experiments to observe the properties of the cuprates. For example, in the past our group has conducted neutron scattering experiments [2] which probe both crystal structure and electron spin properties of the cuprates. X-ray scattering experiments in the near future will provide information on charge ordering in the cuprates and how it relates to superconductivity. All of these methods, along with data from other techniques (such as scanning tunneling microscopy and angle-resolved photoemission spectroscopy) by our collaborators, will aid in progress towards a theory behind high T_c superconductivity.

References

[1] J. G. Bednorz, and K. A. Müller. "Perovskite-type oxides—The new approach to high-Tc superconductivity," Rev. Mod. Phys. 60, 585 (1988).
[2] E. M. Motoyama. "Neutron scattering studies of the electron-doped high-temperature superconductor neodymium cerium copper oxide." Dissertation, Stanford University. 2009.